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LOW TEMPERATURE THERMAL EXPANSION OF VARIOUS MATERIALS

By Henry L. Laquer

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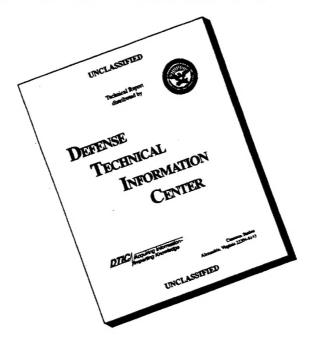
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#### LOS ALAMOS SCIENTIFIC LABORATORY

of the

#### UNIVERSITY OF CALIFORNIA

Report written:
December 9, 1952

LOW TEMPERATURE THERMAL EXPANSION OF VARIOUS MATERIALS

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# LOW TEMPERATURE THERMAL EXPANSION OF VARIOUS MATERIALS

By Henry L. Laquer

#### ABSTRACT

The low temperature thermal expansion of 15 miscellaneous substances has been measured. Literature data for several other materials of possible interest have also been collected. The length changes between 300°K and 0°K are reported in tabular form as an aid in the engineering design of cryogenic devices.

References to the literature on low temperature expansion studies, covering the last 50 years, are given in an appendix. 71 references. (A. In )

#### I. INTRODUCTION

In addition to the measurements on the low temperature thermal expansion of plastics which have been reported in IADC-1230 (rev.) (AECU-2161), (43)\* we have measured the expansion of various metallic and non-metallic materials which were of interest to the Laboratory. These measurements do not represent work of extreme precision, but were made only to obtain engineering information.

<sup>\*</sup> Numbers in parentheses refer to bibliography at end of paper.

#### II. APPARATUS AND PROCEDURE

The apparatus is shown schematically in Fig. 1. It is the same fused quartz tube dial gauge dilatometer mentioned in previous reports (43)(70) and is patterned after the A.S.T.M. standard instrument. (3) The figure should be self-explanatory. After assembly of the specimen in the apparatus, the bell jar (not shown) and outer quartz tube are flushed several times with helium gas and then filled with helium to an absolute pressure of 15 mm of Hg. Dewar vessels containing various refrigerants are raised about the quartz tubes. The refrigerants and their temperatures at the local atmospheric pressure of 580 mm are: boiling hydrogen (19.5°K), pumped nitrogen (65-70°K), boiling nitrogen (75.2°K), dry ice in acetone (190-210°K), ice in salt water (270°K), water at room temperature (295°K), and, sometimes, hot water (320-350°K). The temperature of the specimen is measured to 0.10, but the calibration uncertainty of the thermocouple is as much as 20.50 in some regions of the temperature interval. As soon as the temperature has become steady (approximately 2 minutes after applying a new refrigerant), the relative length of the specimen is recorded to 10-5 inches with a reading uncertainty of  $\pm 1 \times 10^{-5}$  inches. The dial gauge readings are then corrected for the non-linearity of the gauge, a correction which has been obtained by checking the gauge against a set of Johannson blocks, and which should be good to 23 x 10-5 inches for large changes in the dial gauge reading. Next, the length differences, AL', corresponding to various temperature intervals, are obtained and the results of separate measurements are averaged. Then small corrections are applied

for the expansion of the molybdenum cap (omitted in our previous work) and of the fused quartz. The cap has an effective thickness of approximately 1/16" (0.060") and, taking the molybdenum data given below (Table II), one obtains a correction of  $6 \pm 1 \times 10^{-5}$  for the temperature interval from 2980K to 200K. This correction is independent of the length of the specimen. On the other hand, the quartz correction is proportional to the length of the specimen and with a 1-inch specimen, amounts to  $7 \pm 3$ x 10<sup>-5</sup> for the same temperature interval (Table I). The final numerical step involves normalizing the corrected length differences AL, to correspond to specimens of unity length at room temperature or, more exactly, at 273.20K. The probable (R.M.S.) error in this normalized  $\Delta L/L$  is about  $\pm 5 \times 10^{-5}$  for specimens of one inch length, although specimen variabilities may lead to much larger uncertainties. The data reported at 20° intervals are obtained from a smoothed plot of the fractional length changes  $\Delta L/L$ . The percentage errors indicated at the top of each column remain constant for all subsequent entries until a different figure is given. In the case of results obtained with a single specimen, the error is taken as the sum of the previously mentioned  $5 \times 10^{-5}$  plus whatever variability was due to the material. In the case of results obtained from more than one specimen, variation among different samples is also included. Finally, in the case of literature data, the error includes variations among different observers, or among different samples investigated by the same observer.

#### III. RESULTS

#### A. GLASSES

All glasses tend to show hysteresis on thermal cycling. Nevertheless, they are used extensively and their thermal expansions as a class are less than those of any other materials.

# (1) Fused Quartz

The data for "Zeiss" fused quartz between room temperature and liquid hydrogen (Table I) are taken from the measurements of Scheel and Heuse. (56) Their work has been substantiated and extended to liquid helium by Keesom and Doborzynski. (40a,b) It is well known that fused quartz from different sources behaves differently qualitatively, in the location of its density maximum, and quantitatively, in the magnitude of its expansion (cf. review by Souder and Hidnert (63)). Thus the  $\Delta L/L$ between room temperature and liquid nitrogen varies by ±50%; however, Scott (59) has shown that, at least with annealed samples, on further cooling to liquid hydrogen the length differences remain constant, i.e., the AL/L curves run parallel. On the other hand, Dorsey III(12) reported that between room temperature and liquid nitrogen the curves of the expansion coefficients, a, for annealed and quenched samples run parallel. At any rate, the errors indicated for Zeiss fused quartz in Table I include only the uncertainty of the original measurements and of the extrapolation to 0°K. A value of ±50% would perhaps be more appropriate for fused quartz samples of unknown origin.

#### (2) Pyrex

The third column of Table I gives the results of a set of measurements on a 1" Pyrex rod of nominally 5 mm diameter taken from the laboratory stock. It was, in all likelihood, Corning Glass No. 774. The percentage error in these results is quite large since the actual length changes were quite small. There is an indication of a length minimum at about 50°K. Tool and Saunders (65) have suggested that borosilicate glasses of the Pyrex type containing a high percentage of silica are essentially two component systems with one phase being almost pure silica and forming a network which pervades the whole glass. Such a system then might well exhibit a density maximum similar to that shown by fused silica. However, the minimum observed by us is well within the experimental error. It also appeared that the sample contracted about 5% less when cooled directly from room temperature to liquid nitrogen or hydrogen, than when an intermediate stop was made near 2000K. Again, the effect is within the experimental uncertainty. Our room temperature expansion coefficient, a, of  $3.2_5 \times 10^{-6}$  c is in excellent agreement with the range of 3.0 to 3.3 x  $10^{-6}$  c reported by Wichers. et al. (68) The interferometric results of Buffington and Latimer, (7) down to 87°K, on an earlier Pyrex glass (G702-EJ) give a somewhat larger room temperature a of 3.5 x  $10^{-6}$  and their absolute values of  $|\Delta L/L|$  \* are about 5% larger than ours over most of the temperature interval, but agree within 2% at their lowest temperature.

<sup>\*</sup> Note that all comparisons will be made in terms of the absolute value  $|\Delta L/L|$  of the fractional length changes.

# (3) Other Glasses

There have been an appreciable number of measurements on other, mostly Jena, glasses. The last column in Table I summarizes the relatively recent data of Keesom and Doborzyński (40a,b) for Jena glass 2954III. The 3% error indicates the difference in results obtained by an interferometric technique on a small sample and by the vertical comparator of Van Agt and Kamerlingh Onnes (67) on a long tube.

Measurements on Jena Glass 16<sup>III</sup> were made by Kamerlingh Onnes and Heuse, (51) Kamerlingh Onnes and Clay, (52) Scheel, (57) Ebert, (14) and Keesom and Bijl. (39) Jena Glass 59<sup>III</sup> was studied by Henning, (26) Scheel, (57) and Ebert. (14) These last three investigators also studied some Berlin porcelains, the expansions of which are even less than those reported above for Pyrex glass.

The thermal expansions for 20 optical glasses between 80°K and room temperature have been reported in a recent paper by Molby. (47a)

#### B. ELEMENTS

The low temperature thermal expansions of the following, mostly metallic, elements are summarized in Table II: B, Be, Li, Mg, Mo, Rh, Th, and Zn. The data for Be, Mo, and Th are taken from the literature. The other materials were studied in our laboratory. The elements will be discussed in order of increasing expansion. The anomalous results obtained on uranium are discussed separately and are summarized in Table IV.

#### (1) Molybdenum

Molybdenum has the lowest low-temperature expansion of any metal included in this report. The values given in Table II are an average of the interferometric investigations of Erfling II<sup>(16)</sup> to -195.4°C (77.8°K) and of Nix and MacNair II<sup>(49)</sup> to -187.0°C (86.2°K) and have been extrapolated to 0°K on the assumption that the coefficient of thermal expansion, a, would continue to decrease smoothly and to approach 0°K with zero slope. Erfling's results for |ΔL/L| on Eindhoven (probably Philips) material are about 1.5% less than those of Nix and MacNair on Westinghouse Lamp Company molybdenum.

#### (2) Boron

Boron was available in the form of a very brittle cake cemented with 9% B<sub>2</sub>O<sub>3</sub>. The cake had been pressed by the powder metallurgy section of CMR-6. A 0.653" long specimen of approximately square cross section was cut from the cake with an abrasive wheel.

The room temperature expansion coefficient,  $\alpha$ , of our sample is about 11 x 10<sup>-6</sup>/°C whereas Dupuy and Hackspill<sup>(13)</sup> report an essentially constant coefficient of 8.3  $\pm$  .3 x 10<sup>-6</sup> for the range from 20°C to 750°C.

According to Lindemann  $II^{(45)}$  the boric acid anhydride has an expansion coefficient of around 15 x  $10^{-6}$  near room temperature. Thus it appears possible that boron without any binder might contract 20 to 30% less than indicated in Table II.

# (3) Beryllium

The beryllium data in Table II are also based on the interferometric studies of Erfling II (16) between room temperature and 80°K. and have been extrapolated to OOK in the usual manner. Beryllium has a hexagonal close-packed structure and hence one might expect considerable anisotropy in samples prepared by ordinary metallurgical techniques. Fortunately, however, Erfling measured single crystals along the two primary directions as well as coarsely polycrystalline material. It appears that the two crystal expansion coefficients,  $\alpha_{\,\text{\scriptsize M}}$  and  $\alpha_{\,\text{\scriptsize L}},$  do not differ greatly from each other and that their weighted average,  $1/3(\alpha_{11} + 2\alpha_{1})$ , is never less than 97% of the measured  $\alpha$  (polycryst.). This absence of expansion anisotropy in the hexagonal crystal is also shown by the high temperature X-ray investigation of Gordon. (21) His high temperature expansion coefficients tie in quite smoothly with the low temperature ones of Erfling. Finally, the comparator study of Hidnert and Sweeney (28) to -120°C (153°K) agrees with the data reported in Table II to within 1%. The figures are the integral of the weighted average of the single crystal coefficients,  $1/3(\alpha_{ij} + 2\alpha_{\perp})$ , and the error has been taken so as to include Erfling's observations on the polycrystalline sample whose  $|\Delta L/L|$  was larger by about 2.5%.

#### (4) Titanium

Our one titanium sample was 2" long and had been machined from stock rod of unknown origin. Titanium is also hexagonal and hence suspect of directional properties. However, the agreement of our measurements with the literature data is quite good. The results of Erfling III, (18) who investigated two foil samples (to 78°K) which had been rolled from compacted powder, straddle our results and agree to within ±2%. Hidnert (33) measured a sample taken from a cast plate (to 83°K) and here, too, the agreement is within our experimental uncertainty -2% at 83°K and -4% at 193°K. Erfling also measured a wire sample known to have directional properties and obtained  $\Delta$  L/L values larger than ours by 11% (at 80°K).

# (5) Rhodium

Our rhódium sample was 2" long and had been made by slotting two pieces of 2" long, 0.200" wide and 0.015" thick sheet halfway down the middle, inserting them in each other, and silver soldering the assembly at the center.

Again we can make a comparison with the literature data of Erfling II<sup>(16)</sup> and of Valentiner and Wallot.<sup>(66)</sup> The former made measurements down to pumped "nitrogen" (58.1°K) and the latter worked down to liquid air (81.1°K). These two sets of measurements agree with each other to within 0.5%; however, their  $\triangle L/L$  values are about 5% larger than ours. Since it is not clear whether this discrepancy is caused by the fact that our material was rolled sheet, or whether it is due to the silver soldering, our results as well as those of Erfling are included in the table.

# (6) Thorium

The thorium data are taken from the work of Erfling III<sup>(18)</sup> who studied a wire sample of approximately 99.8% purity. The extrapolation of his measurements from 57°K to 0°K has been performed in the usual manner.

Erfling's room temperature  $\alpha$  is 10.9 x 10<sup>-6</sup>/°C. Hidnert and Sweeney (J. G. Thompson<sup>(64)</sup>) have reported a value of 11.1 for the 20°C to 60°C interval. Investigators at Iowa State College <sup>(69)</sup> give a figure of 11.0 x 10<sup>-6</sup> and note that it remains essentially constant over the range from 25° to 600°C.

# (7) Magnesium

A single 1" long magnesium sample was turned down from a  $\frac{1}{4}$ " diameter stock rod. The material is listed in the stock catalog as an alloy (FS-1).

Grüneisen<sup>(22)</sup> measured magnesium down to  $-183^{\circ}$ C (90.2°K) at which temperature our  $|\Delta L/L|$  value exceeds his by about 2.5%. The average values for cast and extruded magnesium measured to about 90°K by Hidnert and Sweeney<sup>(29)</sup> are less than ours by about 9%; however, some of their individual measurements agree with ours to within 1%.

# (8) Zinc

The dilatometric single crystal study of Grüneisen and Goens (23) indicated that zinc is very anisotropic and that the expansion coefficient at right angles to the hexagonal axis is negative below 86°K. Our measurement was made on a 1" long polycrystalline sample taken from a stock item

 $6 \times 1\frac{1}{2} \times 9/16$ " slab, which appeared to have been hot rolled from a billet and subsequently cold sheared. The objective of the measurement was to look for a negative expansion coefficient, and the results reported in Table II are only applicable to our one specific specimen.

The interferometric measurements of Dorsey II<sup>(11)</sup> on cast zinc give  $|\Delta L/L|$  values larger than ours by 10% at 93.2°K. Similarly, the weighted average of the single crystal measurements<sup>(23)</sup> exceeds our  $|\Delta L/L|$ 's by 15% at 90°K and by 20% at 20°K. On the other hand, the  $|\Delta L/L|$  values of Grüneisen, <sup>(22)</sup> and of Lindemann I<sup>(44)</sup> are only about 30% of ours.

# (9) Lithium

Two lithium samples were measured. Both had been extruded in a steel die by CMR-2. The extrusion pressure was less than 250 pounds acting on a 1½" diameter plunger and could not be measured accurately. The samples tarnished slightly, although they were kept under kerosene. They had a diameter of 0.198". The compressional stress of about 15 psi produced by our measuring apparatus was probably responsible for the shortening of the specimens during the course of each run. Thus, the first one decreased in length from 0.782" to 0.774", and the second one from 1.108" to 1.102".

The normalized results,  $\triangle L/L$ , of our measurements are reproduced in Fig. 2 together with some of the data of Simon and Bergmann, (61) who studied a 30 cm long sample in a "thermally symmetrical" fused quartz dilatometer. They covered the range from liquid air (89.4°K) to the ice point and were unable to make measurements at higher temperatures due to

softening of their sample. The agreement between our results and those of Simon and Bergmann is extremely close and well within our estimated experimental error of 1.5%.

# (10) Uranium

The anomalous behavior of the thermal expansion of uranium at low temperatures has been discussed in a previous report (58)(70) by one of the authors. At that time, it was shown that three samples taken along different directions from a 2" diameter casting, which had been annealed for 6 hours in the  $\beta$ -phase and then cooled slowly to room temperature, showed a length minimum and hence a density maximum between 40 and  $50^{\circ}$ K. It was also shown that there did not seem to exist appreciable orientation effects, and, hence, that the anomaly was not caused by directional properties of the samples. Finally, the room temperature value of the coefficient of expansion,  $\alpha$ , was found to be  $13.5 \pm .5 \times 10^{-6/\circ}$ C. This again indicated that our samples were randomly oriented since the average for the three crystal coefficients is  $12.2 \pm 2 \times 10^{-6/\circ}$ C (= 3 x 14.7 x  $10^{-6/\circ}$ C). (36)

attacked from three different directions. First, a literature survey was undertaken to discover whether any materials other than fused quartz exhibited similar density maxima and how such maxima could be explained. Second, an attempt was made to modify our dilatometer to obtain temperature control in the region of the density maximum in order to measure the complete dilatation curve. Third, more samples were obtained, some of

which were sent to Professor H. L. Johnston at Ohio State University for a detailed interferometric investigation.

efficients along certain crystal axes do occur, and that, indeed, there are a few materials which exhibit negative volume coefficients. Table III lists all the materials known to exhibit such an anomaly. The existence of negative coefficients along certain crystal axes had actually been predicted by Grüneisen and Goens (23) on the basis of "semitheoretical" considerations. However, the density maxima have not been explained, except perhaps in the case of fused quartz which is considered a two component system (Sosman (62)). The real puzzles are the substances which crystallize in the cubic system, such as sphalerite (ZnS), silicon, and type 304 stainless steel. On the other hand, all of these materials can exist with alternate structures, and, hence, retarded phase changes cannot be excluded entirely.

The unsuccessful attempt to modify the dilatometer for accurate measurements at intermediate temperatures, especially between 20° and 60°K, consumed about 2 man-months. A heater was wound on a copper block which surrounded the quartz tubes near the sample, and the whole assembly was vacuum jacketed. We succeeded in varying the temperature quite gradually and satisfactorily. However, in almost all runs the dial gauge appeared to stick below 60°K. Our most likely explanation has been that there were sufficient impurities in the helium exchange gas which, during the relatively long times involved in these measurements, could diffuse to the coldest spot and solidify there, thus "gumming up" the dilatometer. Unfortunately, the whole apparatus had been improvised,

and time and space limitations were such that we could not set up a vacuum bench for the purification of the exchange gas. Hence, the detailed measurements in the 20° to 60°K range were abandoned.

All the new samples of uranium were taken from a 12" o.d., 8" i.d. cast ring, which had been cooled slowly but which had not been given any further heat treatment. The three specimens in set A were 1" long and had a diameter of 0.200". The three specimens in set C were originally 0.200" long and had a diameter of  $\frac{1}{2}$ ". They were machined further and quartered (Dwg. 11Y-31347B-2) so as to be suitable for an interferometric study at Ohio State University (0. S. U.). In each set, the specimens had the following orientations (Dwg. 11Y-31347C-1):

No. 1 had its axis, i.e., the direction along which the dilatation was to be studied, in the plane of the large ring and parallel to its radius.

No. 2 had its axis in the plane of the ring and at right angles to its radius.

No. 3 had its axis perpendicular to the plane of the ring and at right angles to its radius.

The results of these investigations were quite surprising in that the minimum was entirely absent with one of our samples (A-2). On the other hand, the O. S. U. sample with the same orientation (C-2) showed the most pronounced minimum. In Fig. 3 we have re-plotted the O. S. U. expansion coefficients, a, from their preliminary graphs. It is to be noted that this figure does not do justice to the accuracy of their experimental work. However, the qualitative features are apparent

in that  $\alpha$  becomes negative at 47  $\pm$  1°K for all three samples, and that, although the expansion coefficients vary appreciably near the region of the anomaly, they are fairly constant near room temperature (16 to 17.5  $\times$  10<sup>-6</sup>/°C). There is some scatter of the experimental points for C-1 between 80° and 100°K, and for C-3 between 80° and 140°K. Our own measurements of  $\Delta$  L/L for A-1, A-2, and A-3 are shown together with the average curve taken from the previous report<sup>(70)</sup> in Fig. 4. Finally, Fig. 5 shows the integrated 0. S. U. results and Table IV lists the smoothed  $\Delta$  L/L averages. It should be stated again that the data reported as measured at 0. S. U. were taken from their preliminary graphs and may well differ from the data that will appear in their final report on this subject.

The only definite statement one can now make about the low temperature thermal expansion of uranium is that the length minimum, when present, occurs at 47 ± 2°K, and that, if it is caused by directional properties, these are small scale rather than large scale ones. We intend to do further work on specimens known to have preferred orientation and also on some fine grained and perhaps alloy samples.

#### C. COMPOUNDS

Only two chemical compounds were investigated. They are lithium hydride and sodium fluoride. Literature data for lithium fluoride are also included in Table V.

# (1) Lithium Fluoride

The lithium fluoride data are taken from the work of Adenstedt(1) who investigated crystalline material with an interferometric technique between the ice point and liquid hydrogen. Henglein's II<sup>(25)</sup> results obtained by direct density determinations at -79°C (194.2°K) and -184°C (89.2°K) give somewhat larger |\Delta L/L| values, 5% at the former temperature, and 10% at the latter.

# (2) Lithium Hydride

Four samples of lithium hydride were investigated. CMR-2 compacted 20 mesh powder at  $400^{\circ}$ C and 20,000 psi in an Aquadag lubricated steel mold. After being held at temperature and pressure for about three minutes, the pressed pellets were allowed to cool in situ to  $200^{\circ}$ C, which required about 30 minutes, and then transferred (in the open air) to a calcium chloride desiccator. The pellets which had a  $\frac{1}{2}$  diameter and varied in length from 1 to  $1\frac{1}{2}$  were machined to the final dimensions within a week of the measurements. The sample lengths varied from about  $\frac{1}{2}$  to  $1^{\circ}$  and all specimens had a diameter of 0.200° except for No. 5 which had a diameter of 0.483° and which was studied in a new and slightly different dilatometer.

The somewhat erratic results obtained in this study are shown in Fig. 6. Not only did measurements on different samples vary

by ±10% from an average, but also subsequent low temperature cycles on the same sample did not reproduce previous ones, especially if a high temperature (+150°C) cycle had intervened. Thus the figures given in Table V are afflicted with the large probable error of ±10%. Nevertheless, with this wide a margin, all the experimental points are included. We cannot state at present whether these variabilities are caused by the method of fabrication or by carelessness on our part in exposing the samples to atmospheric moisture, or whether they are an intrinsic property of slightly contaminated lithium hydride.

Some expansion measurements on LiH were also made at elevated temperatures (+200°C). They are not included here because they appeared erratic and because other investigators were working on the problem. However, their repeated attempts to carry measurements to higher temperatures also gave inconsistent results. One is thus left with the strong suspicion that a permanent change takes place in the material at or above 200°C.

# (3) Sodium Fluoride

The sodium fluoride samples were machined from a  $2\frac{1}{4}$ " dia.  $\frac{1}{2}$ " thick cake pressed by the powder metallurgy section of CMR-6. Specimen No. 1 had a length of 1.009", specimen No. 2 had a length of 0.571". The results obtained from these two samples agreed to within 1.5%. A comparison can again be made with the direct density determinations of Henglein II (25). The  $|\Delta L/L|$  values calculated from his work are about 8 to 10% larger than ours.

#### D. FILLED POLYMERS

The thermal expansion of various commercial polymers has been reported elsewhere. (43) The expansion of polythene filled with varying amounts of inorganic oxides will be discussed here.

Such filled or loaded polymers offer an opportunity to introduce materials with desirable nuclear properties but available only as powders. At the same time the concentration of these materials can be varied over wide ranges.

Ferric oxide, Fe<sub>2</sub>O<sub>3</sub> (jeweler's rouge), was chosen as a typical finely divided and readily dispersed oxide and samples containing 25, 50, and 75 volume percent in polythene were investigated in order to determine whether the expansion behaved in a simple (additive?) manner.

They were molded for ten minutes at 150 to 165°C under a pressure of 2000 psi and then allowed to cool in the mold with the pressure maintained. Most of the Fe<sub>2</sub>O<sub>3</sub> samples were molded into disks of 3" diameter and 3/4" thickness except two (194-1 and 194-2) which were molded into 1-1/2" long cylinders of 3/4" diameter. The scandium and germanium oxide samples were also molded into 3/4" diameter cylinders about 1" long. The densities of the molded pieces were determined by CMR-6, and the probable compositions, as reported in Table VI, were calculated on the assumption of a linear relationship between composition and density. In general, the agreement

between nominal and probable composition is reasonably good except for the 75% Fe<sub>2</sub>O<sub>3</sub> samples. The low values for the density of these samples have been checked and are definitely not caused by experimental errors. They can be explained in two ways. It is possible either that the samples were not completely compacted and had microscopic voids, or that there might be some sort of interaction between the filler and the polythene. Filler reinforced polythene samples have been mentioned in the literature (cf. Bostwick and Carey<sup>(6a)</sup>) and there has been a discussion of the thermodynamics of filler reinforcement (Rehner<sup>(52a)</sup>). However, to the best of our knowledge, there have not been any studies on Fe<sub>2</sub>O<sub>3</sub> reinforced polythene, and there is no way to predict whether or not there should be a simple relation between density and composition. Table VI also lists the dimensions of the machined pieces. The machining operation proved to be a somewhat difficult job especially with the 75% Fe<sub>2</sub>O<sub>3</sub> samples.

Table VII summarizes the fractional length changes for the various samples as well as for pure polythene. (43) It is apparent that, with the exception of 187-A, samples taken from the same molded piece do not vary greatly. However, samples from different moldings, although of nearly constant density (194-1, 194-2, and 194-3) can vary by 15%. The average values reported are straight averages except with the 75% Fe<sub>2</sub>O<sub>3</sub> samples. where the longer (b) sample has been weighted four times as heavily as the shorter (a) sample. It is quite clear from the data that variabilities among nominally identical samples can be appreciable and that with the present methods of sample preparation and dispersion, one cannot expect

results to be reproducible by much better than 10%. Nevertheless, it is apparent that the overall thermal expansion  $|\Delta L/L|$  is less than what one would expect if a linear law of mixtures held true. In other words, there seems to be an appreciable interaction making the mixtures much more like the oxide than would correspond to the relative amounts of material present, and it seems plausible that the attainment of such a structure would be accompanied by a lowering of the density. The same facts are shown in Fig. 7 where we have plotted the coefficients of linear thermal expansion for the various polythene - Fe<sub>2</sub>03 mixtures. The value for the room temperature expansion coefficient of Fe<sub>2</sub>O<sub>3</sub> has been taken from the work of Fizeau, as reported by Dane (8a). At 300°K the coefficients of expansion of the 25% Fe<sub>2</sub>0<sub>3</sub> mixtures are about 80%, those for the 50% Fe<sub>2</sub>0<sub>3</sub> mixtures about 45%, and those for the 75% Fe<sub>2</sub>O<sub>3</sub> mixtures about 40% of what one would calculate from a linear law. At 100°K the corresponding percentages are 83, 48, and 65(?)%. However, before making any further speculations on the problem of the oxide-polymer interaction, the compositions of the samples should be determined by an independent method.

#### IV. CONCLUSIONS

From the data presented in this report it appears that for most engineering materials length changes,  $\triangle L/L$ , between room temperature and low temperatures can be known with an accuracy of  $\pm 5 \times 10^{-5}$  or better. Assuming then that room temperature dimensions are obtainable with any required accuracy, low temperature dimensions will be known with the above accuracy; i.e., a structure of one inch will be known to within 0.0001", a 10" structure to within 0.001" and a 100" structure to within 0.01". Such accuracies are certainly within the limits of good engineering practice.

On the other hand, for non-cubic and strongly anisotropic materials, which are inherently poorer from an engineering standpoint, the dimensions at low temperatures may be uncertain by five to ten times the amounts stated above. Even if the single crystal expansion coefficients are known accurately, the expansion of a polycrystalline sample of the material may well be different from their weighted average because of the development of directional properties during the process of fabrication.

From the literature references, which are collected in Appendix B, one sees that measurements have been made on almost all common metals at least to the temperature of liquid air or nitrogen. The extrapolation of these measurements to lower temperatures will not introduce excessive errors, especially since one can be guided by the parallelism between the variation of the heat capacity,  $C_p$ , and the coefficient of thermal expansion, a. The deviations from this empirical Grüneisen relationship are always small and are only shown by measurements of much higher precision than was attempted in the present engineering study. The expansion

information is also available for many commonly used alloys, but is much less complete for most of the non-metals and salts, except for the alkali halides.

Finally, if there is a question as to the low temperature expansion of almost any mixture, one can obtain reasonably complete information from dilatometric observations at room temperature, the ice point, dry ice, liquid nitrogen, and pumped nitrogen. Extrapolation of such measurements to lower temperatures can be attempted with confidence and is not likely to introduce errors any greater than the variabilities inherent in poorly and difficultly defined materials.

#### APPENDIX A

# UNPUBLISHED OHIO STATE UNIVERSITY MEASUREMENTS

Through the courtesy of Professor Herrick L. Johnston of Ohio State University, we have had made available to us precision results on the expansion coefficients of various materials<sup>(2)</sup> which might be used in the construction of cryogenic equipment. The coefficients were reported at 10° intervals between 0 and 300°K. We have integrated these results by assuming that the coefficient was constant over each ±5° interval and simply summing the coefficients (times ten). The data reported in Table VIII do not do justice to the precision of the original work, but are sufficient for our purposes.

#### APPENDIX B

#### LITERATURE SURVEY

In collecting the information presented in this report, a survey of the literature on low temperature expansion studies was made, based on Science Abstracts. An attempt has been made to include all original investigations covering temperatures below those of "dry ice."

Table IX lists the elements of the periodic table in order of atomic number and gives references to the thermal expansion literature together with the lowest temperature reached in each study. The method of investigation is indicated in the bibliography by the abbreviations: IN, DIL, COMP, DEN, and X-ray. A brief discussion of these five basically different methods and their principal users may be of some interest:

(1) The interferometer (INT) was first used for room temperature expansion studies by Fizeau. It was adapted to low temperature work by Scheel (54-57) at the Reichsanstalt, and by Ayres (5) and Dorsey (10-12) at Cornell University. It was also used by Valentiner and Wallot, (66) Disch, (9) Buffington and Latimer, (7) and very extensively by Grüneisen's students at Marburg University (Adenstedt, (1) Erfling, (16-18)) as well as by Nix and MacNair (48-49) at the Bell Labs. The most recent and still operating low temperature interferometer is installed at Ohio State University. (2)

Interferometric studies can be made either on an absolute or on a relative basis. Their advantages are the small specimen length (1 cm)

and the high precision that is attainable. However, the method is not a simple or quick one.

(2) The fused quartz or glass tube dilatometer (DIL) with microscopic observation was first described by Henning (26) and was used pretty much in its original form by Grüneisen, (22) Lindemann, (44,45) Grüneisen and Goens, (23) Ebert, (14) and Simon and Bergmann. (61) A slightly different form of optical indication was used by Borelius and Johansson, (6) Krupkowski, (41,42) and by the Japanese investigators. (4,46,50) The dial gauge modification is primarily the work of Hidnert and Sweeney (29) at the National Bureau of Standards and has been made a standard method by the American Society for Testing Materials. (3)

Dilatometric studies have to be relative measurements. In order to attain reasonable precision, the sample length has to be at least one inch. If one is willing to settle for measurements at a few fixed points, as was done in our work, then the dilatometer offers a very convenient and relatively quick method.

(3) Absolute expansion measurements can be made by direct length comparisons. Such comparators (COMP) can take various forms, but they all utilize long specimens (1 m) whose length at various temperatures is compared with that of an approximately equal length of a thermostated standard. The comparison is made with traveling microscopes.

The vertical comparator (vert COMP) is a product of the Leiden cryogenic institution. (67,37-40a) It was developed from the earlier studies of H. K. Onnes and others (51,52) where a one meter rod was almost completely immersed in a refrigerant and a correction was applied for the gradients

along its exposed ends. In the vertical comparator, the material under investigation forms the inner dewar wall and the length of a marked one meter long portion is compared with a fixed length on the outer dewar jacket which is kept at a constant temperature. The only drawback of this method is that it requires the construction of very special inner dewar vessels.

The horizontal comparator (hor COMP) was developed at the Bureau of Standards by Hidnert, Schad, and Souder (53a,b,63) It requires a large stirred bath for low temperature work, and has not been used at temperatures below 130°K. The primary use of the horizontal comparator has been in high temperature studies where the bath is replaced by a furnace.

- (4) Density determinations (DEN) by immersion weighing in liquid air were described by Dewar, (8) and by Grüneisen and Sckell. (23a) Henglein (24,25) obtained the density by finding the volume of liquid or gas displaced at low temperatures. These methods appear rather difficult to engineer satisfactorily.
- (5) X-ray investigations (X-ray) are probably the most fundamental way to attack the problem of low temperature thermal expansion. Their main drawback has been the requirement of long exposure times, which, however, could be materially reduced with modern counter spectrometers. Only a few references to X-ray work are included in this review, which is quite incomplete in this respect.

Table X gives references to low temperature studies on various alloys.

Table XI gives the corresponding information for a number of inorganic compounds.

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TABLE I
Thermal Expansion of Glasses

		$\Delta L/L \times 10^5$	
(°K)	Zeiss Fused Quartz	Pyrex 774	Jena 2954 <b>1</b> 11
0	+9.6 ± 3%	-48.5 ± 10%	-91.5 ± 3%
20	7.41 ± 0.1%	49.5	90.0
40	5.71	50.5	88.5
60	3.83	50.0	86.0
80	2.22	47.5	83.0
100	+0.93	44.0	79-0
120	-0.06	40.0	73.0
140	0.80	35.5	66.0
160	1.27	31.0	57.5
180	1.51	26.0	48.5
200	1.55	21.0	39.0
220	1.37	15.5	29.0
240	1,00	9.5	18.5
260	-0.45	-4.0	-7.5
273.2	0	0	o
280	+0.27	+2.0	+4.0
300	1.10	8.5	15.5
3.20	2,05	15.0	27.5
340	3.12	21.5	39.5
360	4.27	28.0	52.5

TABLE II

Thermal Expansion of Some Elements

							-	57-					
T4+birm	(H and L)	-698 ± 1.5%	269	692	682	658	623	578	522	458	387	311	230 146 -59 0 +31 122
7400	(H and L)	-500 ± 2%	664	167	472	443	7,07	367	324	279	233	18%	137 87 -35 0 118 72
	Magnesium (H and L)	1		430				348	311	27.1	227	181	132 82 -32 0 118 68
	Thorium (Lit.)	-223 ± 2%	223	219	209 ± 1%	194.5	177.5	159	139.5	119.5	66	78	57 35.5 -14 0 +7.5 29
105	Rhodium L) (Erfling II)	8 -144 ± 0.5%	143.5	742	138.5	132.4	123.5	भूता	,001	87	22	28.	26.5 26.5 -10.5
$\Delta I/L \times 10^5$	R (H and L	-142 ± 3%	141	137	131	721	ñ	105	8	: 젊	89	太	3 % 2 0 9 8
	Titanium (H and L)	-132 ± 3%	132	131	130	356	श्रम श्रम	308	6	85	2	× 88	324028
	Beryllium (Literature)	-107.5 = 3%	107 .	107	2001	0.101	105.0 ± 2%	, 101	8	88	78.7	65.0	50.0 32.5 -13.5 0 +7.5 29.0
	Boron+9£BO <sub>3</sub> (H and L)	-104 ± 58	10,	13	3 5	100 + 20	96	<b>1</b> 0	2 8	2 ₹	÷ ;	6.8	45 32 -13 -13 29
	Molybdenum (Literature)	-85 K + 1%	1 ( ) ( )	7.00	\$ 8	83.2	75.0		8.00	01.7	25.7	45.0 35.8	
-	OK)	c	> 8	3 5	3 (	8	8 8	3	2 5	9 5	200	28 PS	220 240 260 273.2 280

TABLE III

Materials with Anomalous Thermal Expansions

Material	Oheemton	Tempe	Temperatures below which a < 0 (OK)	v which α <	0 (OK)
		"avg.	a d	- 1884	70
Fused Quartz: Heräus Zeiss Silicate Co.	Scheel, 1907(55) Scheel, 1907(55) Scheel and Heuse, 1914(56)	227 189 205	X XX		XXX
Si (fused)	Valentiner and Wallot, 1915(66)	971	XX		XX
Zn crystal (hexagonal)	Grüneisen and Goens, $1924(23)$	None	None		%
ZnS, sphalerite (cubic)	Adenstedt, $1936^{(1)}$	19	XX		X
Calcite (hexagonal)	Adenstedt, 1936 <sup>(1)</sup>	101	None		Above R.T.
Beryl (hexagonal)	Erfling II, 1939 (16)	262	Above R.T.		188
Si crystal (cubic)	Erfling III, 1942(18)	143	XX		X
304 Stainless (cubic)	Altman, Rubin and Johnston <sup>(2)</sup>	33	XXX		X
			a(100)	°b(010)	a <sub>c</sub> (001)
U (orthorhombic)	Schuch and Laquer (58,70)	40-50		(96)002	
			1		

TABLE IV

Thermal Expansion of Uranium

T			Δ	L/L x 10	5		
(°K)	LAMS-1358	A-1	A-2	A-3	C-1	C-2	C-3
0	-234	-241	-269	-158	-259.5	-264.0	-250.5
20	238	245	266	162	267.1	269.4	255.6
40	275	290	255	235	302.5	324.3	272.0
60	269	284	240	236	301.8	329.9	268.0
80	253	262	223	215	281.0	309.6	251.1
100	231	237	204	193	256.1	283.1	230.1
120	206	211	184	170	229.3	253.4	207.2
140	180	184	163	148	201.4	222.1	183.2
160	153	157	140	126	172.7	189.9	158.2
180	126	129	117	104	143.2	157.2	132.3
200	99	101	94	82	113.2	124.1	105.5
220	72	73	69	59	82.6	90.6	77.9
240	45	46	44	37	51.8	56.8	49.4
260	-18	-18	-18	-15	-20.7	-22.6	-20.0
273.2	0	0	0	0	0	0	0
280	+9	+ 9	+ 9	+ 7	+10.7	+11.8	+10.6
300	36	36	36	30	42.4	46.4	42.2
320			63	52			
340			90	74			
360			117	96			
380			144	118			

Note: No average values are reported because wide variation among samples taken from same casting causes an uncertainty of ±20%.

TABLE V

Thermal Expansion of Some Inorganic Compounds

T		$\Delta L/L \times 10^5$	
(°K)	Lif	Lih	NaF
0	-434 + 0.5%	-390 ± 10%	-480 ± 1%
20	433	390	479
40	431	389	476
60	427	386	469
80	420	379	457
100	405	367	436
120	382	349	406
140	349	324	368
160	309	293	323
180	264	256	273
200	215	212	218
220	160	162	160
240	103	106	100
260	- 43	- 44	- 40
273.2	0	• 0	0
280	+ 23	+23	+21
300		95	83

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TABLE VI

Description of Filled Polythene Samples

Nominal Composition	Density	Probable Composition	D <b>i</b> mensions
25% Fe <sub>2</sub> O <sub>3</sub>	2,00	25.0	0.9995 x 0.203
"	1.99	25.0	0.4580 x 0.204
Ħ	1.97	24.0	0.981 <sub>0</sub> x 0.205
11	1.97	24.0	$1.0335 \times 0.206$
50% Fe <sub>2</sub> 0 <sub>3</sub>	2.92	46.5	0.961g x 0.205
n	2.92	46.5	$0.952_2 \times 0.20_7$
75% Fe <sub>2</sub> 0 <sub>3</sub>	2.80	43.5 ?	0.683 <sub>0</sub> x 0.217
11	2.87	45.0 ?	$1.002_5 \times 0.214$
,50% Sc <sub>2</sub> 0 <sub>3</sub>	1.96	35.5 37.0	0.7975 x .209 0.8111 x .205
50% GeO <sub>2</sub>	2.49	41.0	0.802 <sub>0</sub> x .206 0.794 <sub>2</sub> x .206
	Composition  25% Fe <sub>2</sub> O <sub>3</sub> "  50% Fe <sub>2</sub> O <sub>3</sub> 75% Fe <sub>2</sub> O <sub>3</sub> "  75% Se <sub>2</sub> O <sub>3</sub> "	Composition Density  25% Fe <sub>2</sub> O <sub>3</sub> 2.00  1.99  1.97  1.97  50% Fe <sub>2</sub> O <sub>3</sub> 2.92  2.92  75% Fe <sub>2</sub> O <sub>3</sub> 2.80  2.87  50% Sc <sub>2</sub> O <sub>3</sub> 1.96  2.01	Composition         Density         Composition           25% Fe <sub>2</sub> O <sub>3</sub> 2.00         25.0           1.99         25.0           1.97         24.0           1.97         24.0           50% Fe <sub>2</sub> O <sub>3</sub> 2.92         46.5           75% Fe <sub>2</sub> O <sub>3</sub> 2.80         43.5 ?           2.87         45.0 ?           50% Sc <sub>2</sub> O <sub>3</sub> 1.96         35.5           2.01         37.0           50% GeO <sub>2</sub> 2.49         41.0

TABLE VII

Thermal Expansion of Filled Polythene Samples

50% GeO <sub>2</sub> 187-A-1 167-A-2	-700 ± 2% 695 685 685 665 635 600	560 515 465 405 340	265 175 - 70 + 35 145
50%   187-A-1	-920 ± 25 915 900 875 840 800	755 700 640 570 485	380 255 -105 0 + 55 215
50% Sc203 198-A wt'd. Avg	-695 ± 3% 690 675 650 620 580	535 485 430 370 305	235 155 - 65 0 + 30 125
75% Fe <sub>2</sub> 0 <sub>3</sub> 192-1 *t'd. AVR	-300 ± 5% 298 292 283 270 254	234 212 187 159 128	95 60 -25 111 47
/L x 10 <sup>5</sup> 50½ Fe <sub>2</sub> 0 <sub>3</sub> 193-1 Avg	-593 ± 2½ 591 580 564 544 523	497 464 421 365 299	224 142 - 57 - 57 0 + 29 115
	-1200 ± 25 1195 1175 1145 1105	995 925 845 750 640	510 340 -140 + 70 285
25% Fe203 194-2	-1395 ± 1% 1390 1365 1320 1270 1210	1145 1070 985 885 760	600 400 -160 0 + 90 350
194-1	-1285 ± 1% 1280 1260 1225 1180 1125	1060 990 905 805 685	535 355 -150 0 + 75 305
Pure (43)	-2090 ± 5% 2080 204.5 1990 1920 183.5	1730 1605 14.55 1280 1080	840 560 -235 -120 190
(NK)	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	120 140 160 130 200	220 240 260 273 .2 280 300

TABLE VIII

## INTEGRATED THERMAL EXPANSION DATA

(H. W. Altman, T. Rubin, and H. L. Johnston, Ohio State University, Unpublished Results, 1949 - 1951)

	ΔL/L <b>x</b> 10 <sup>5</sup>					
Material	O°K	<b>2</b> 0°K	75°K	105°K	<b>2</b> 05 <b>°</b> K	295°K
410 Stainless Steel	0	0.05	5.7	17.5	91.2	178.3
1020 Low Carbon steel	0	0.1,	8.,	22.4	105.5	203.9
Inconel	0	0.02	9•9	26.9	122.4	231.5
99.6% L-Nickel	0	0.03	9•5	26.8	122.6	232.1
Contracid	0	0.04	9•8	27.4	124.6	234.7
Cold Rolled Monel	0	0.13	12.5	32.4	136.4	254.0
304 Stainless Steel (18-8)	0	-0.94	10.7	34.9	158.6	296.3
O. F. H. C. Copper	0	0.17	20.1	48.5	184.4	329 • 2
Free Turning Yellow Brass	0	0.34	28.8	64.3	223.2	388.4
99.99% Aluminum	С	0.13	19.2	50.0	216.2	404.7
99.99% Lead	0	9.4	120.0	194.9	461.2	715.5

Note: Integration performed by simple summation. Original data contain one more significant figure.

TABLE IX

The Flements

Atomic Number	Element	T min*	Method	Reference
3	Lithium "	89 20	DIL	Simon and Bergmann <sup>61</sup> This report p. 15
4	Beryllium " (crystal) "	153 90 80	COMP INT INT	Hidnert and Sweeney <sup>28</sup> Erfling II <sup>16</sup> Erfling II <sup>16</sup> This report p. 12
5	Boron	20	DIL	This report p. 11
6	Carbon (Diamond) " (Graphite)	85 78	INT INT	Dembowskas (ref. Grüneisen <sup>22a</sup> ) Erfling IIlo
11	Sodium	84 80	DEN DIL	Dewar <sup>8</sup> Siegel and Quimby <sup>60</sup>
12	Magnesium "" ""	90 90 20 20	DIL DIL, COMP DIL DIL	Grüneisen <sup>22</sup> Hidnert and Sweeney <sup>29</sup> Ebert 14 This report p. 14
13	Aluminum  " " " " " " "	93 82 20 87 20 82 15	INT DIL DIL INT DIL INT INT	Ayres <sup>5</sup> Henning <sup>26</sup> Lindemann I <sup>44</sup> Buffington and Latimer <sup>7</sup> Ebert <sup>14</sup> Nix and MacNair I <sup>48a</sup> O. S. U. <sup>2</sup> (App. A)
14	Silicon " (crystal)	82 86 78	INT DIL INT	Valentiner and Wallot <sup>66</sup> Simon and Bergmann <sup>61</sup> Erfling III <sup>18</sup>
15	Phosphorus			
16	Sulfur			

<sup>\*</sup>T min = Lowest temperature reached in investigation.

TABLE IX (continued)

Atomic Number	Element	T min	Method	Reference
19	Potassium			
20	Calcium	57	INT	Erfling III <sup>18</sup>
21	Scandium			
22	Titanium "	78 83 20	INT DIL DIL	Erfling III <sup>18</sup> Hidnert <sup>33</sup> This report p. 13
23	Vanadium	58	INT	Erfling III <sup>18</sup>
24	Chromium "	83 57 168	INT INT COMP	Disch <sup>9</sup> Erfling II16 Hidnert <sup>32</sup>
25	Manganese "	83 90 78	INT INT INT	Disch <sup>9</sup> Erfling III6 Erfling IIa <sup>17</sup>
26	Iron " " " "	93 20 90 20 92	INT DIL DIL INT INT	Dorsey I <sup>10</sup> Ebert <sup>14</sup> Simon and Bergmann <sup>61</sup> Adenstedt <sup>1</sup> Nix and MacNair I <sup>48a</sup>
27	Cobalt			
28	Nickel " " " " " " " "	82 83 20 90 78 77 81 15	DIL INT DIL DIL INT DIL INT	Henning <sup>26</sup> Disch <sup>9</sup> Krupkowski and De Haas <sup>41</sup> Simon and Bergmann <sup>61</sup> Adenstedt <sup>1</sup> Aoyama and Ito <sup>4</sup> Nix and MacNair I <sup>48a</sup> O. S. U. <sup>2</sup> (App. A)
29	Copper  II  II  II  II  II  II	82 93 20 103 109 20	DIL INT DIL INT COMP	Henning <sup>26</sup> Dorsey I <sup>10</sup> Lindemann I <sup>44</sup> Borelius and Johansson <sup>6</sup> Buffington and Latimer <sup>7</sup> Keesom, et al <sup>37</sup>

TABLE IX (continued)

Atomic Number	Element	T min (°K)	Method	Reference
29	Copper " " " " " " " " " "	20 90 89 77 88 ——————————————————————————————	DIL DIL INT DIL LIT	Krupkowski and De Haas <sup>41</sup> Simon and Bergmann <sup>61</sup> Adenstedt <sup>1</sup> Aoyama and Ito <sup>4</sup> Nix and MacNair I <sup>48a</sup> Gaumer and Scott <sup>20</sup> Rubin, Altman and Johnston <sup>71</sup> (App. A
30	Zinc " (crystal) " (crystal) " (crystal)	93 90 20 20 78 ? 20	INT DIL DIL DIL X-ray DIL	Dorsey I <sup>10</sup> Grüneisen <sup>22</sup> Lindemann I <sup>44</sup> Grüneisen and Goens <sup>23</sup> McLennan and Monkman <sup>47</sup> This report p. 14
31	Gallium			
32	Germanium			
33	Arsenic			
34	Selenium	93	INT	Dorsey II <sup>11</sup>
37	Rubidium	90	X-ray	Hume-Rothery and Lonsdale35
38	Strontium			
39	Yttrium			
40	Zirconium "	60 78	INT IN <b>T</b>	Erfling II <sup>16</sup> Erfling III <sup>18</sup>
41	Niobium *	138 61	UIT INT	Hidnert and Krider <sup>31</sup> Erfling III <sup>18</sup>
42	Molybdenum " "	131 83 86	COMP INT INT	Schad and Hidnert <sup>53a,b</sup> Disch <sup>9</sup> Nix and MacNair II <sup>49</sup> This report p. 11
43	Masurium			

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TABLE IX (continued)

Atomic Number	Element	T min	Method	Reference
44	Ruthenium			
45	Rhodium	81 58 20	INT INT DIL	Valentiner and Wallot <sup>66</sup> Erfling II <sup>16</sup> This report p. 13
46	Palladium "	82 83 86	DIL INT INT	Henning <sup>26</sup> Scheel <sup>54</sup> Nix and MacNair II <sup>49</sup>
47	Silver  n  n  n  n  n  n	93 82 93 20 87 20 20 87	INT DIL INT DIL INT COMP DIL INT	Ayres <sup>5</sup> Henning <sup>26</sup> Dorsey I <sup>10</sup> Lindemann I <sup>44</sup> Buffington and Latimer <sup>7</sup> Keesom, et al <sup>38</sup> Ebert <sup>14</sup> Nix and MacNair II <sup>49</sup>
48	Cadmium " (crystal) " (crystal)	93 90 20 78 ?	INT DIL DIL Xray	Dorsey I <sup>10</sup> Grüneisen <sup>22</sup> Grüneisen and Goens <sup>23</sup> McLennan and Monkman <sup>47</sup>
49	Indium	83	DIL, COMP	Hidnert and Blair <sup>34</sup>
50	Tin " (crystal)	93 90 58	INT DIL INT	Dorsey I <sup>10</sup> Grüneisen <sup>22</sup> Erfling II <sup>16</sup>
51	Antimony " (crystal)	93 90 58	INT DIL INT	Dorsey I <sup>10</sup> Grüneisen <sup>22</sup> Erfling II <sup>16</sup>
52	Tellurium			
53	Iodine	84	DEN	Dewar <sup>8</sup>
55	Cesium			
56	Barium			

TABLE IX (continued)

Atomic Number	Element	Tomin	Method	Reference
57 58	Lanthanum Cerium	78 <b>78</b>	DIL	Trombe and Foex 64a Trombe and Foex 64a, b
72	Hafnium			
73	Tantalum	195 83 92	INT DIL, COMP INT	Disch <sup>9</sup> Hidnert <sup>30</sup> Nix and MacNair II <sup>49</sup>
74	Tungsten "	83 173	INT COMP	Disch <sup>9</sup> Hidnert and Sweeney <sup>27</sup>
75	Rhenium			
76	Osmium			
77	Iridium "	90 81	DIL RIT	Grüneisen <sup>22</sup> Valentiner and Wallot <sup>66</sup>
78	Platinum " " " "	91 82 83 93 81 85	COMP DIL INT INT INT	Onnes and Clay <sup>52</sup> Henning <sup>26</sup> Scheel <sup>54</sup> Dorsey I <sup>10</sup> 'Valentiner and Wallot <sup>66</sup> Nix and MacNair II <sup>49</sup>
79	Gold " "	93 90 20 86	INT DIL DIL INT	Dorsey I <sup>10</sup> , II <sup>11</sup> Grüneisen <sup>22</sup> Ebert <sup>14</sup> Nix and MacNair I <sup>48</sup> a
80	Mercury " (crystal) " (crystal)	84 85 83	DEN DIL, DEN DIL	Dewar <sup>8</sup> Grüneisen and Sckell <sup>23a</sup> Hill <sup>34a</sup>
81	Thallium (crystal)	57	INT	Erfling II <sup>16</sup>
82	Lead " " " " "	93 90 20 20 85 15	INT DIL DIL DIL INT INT	Dorsey II <sup>11</sup> Grüneisen <sup>22</sup> Lindemann I <sup>44</sup> Ebert <sup>14</sup> Nix and MacNair II <sup>49</sup> O. S. U. <sup>2</sup> (App. A)

TABLE IX (concluded)

Atomic Number	Element	T min	Method	Reference
83	Bismuth " (crystal)	93 90 20	INT DIL INT	Dorsey I <sup>10</sup> Grüneisen <sup>22</sup> Erfling II <sup>16</sup>
84	Polonium			
88	Radium			
89	Actinium			
90	Thorium	<i>5</i> 7	INT	Erfling III <sup>18</sup> This report p. 14
91	Protactinium			
92	Uranium	20	DIL	This report p. 16
93	Neptunium			
94	Plutonium	93	DIL	Elliott and Tate <sup>69a</sup>

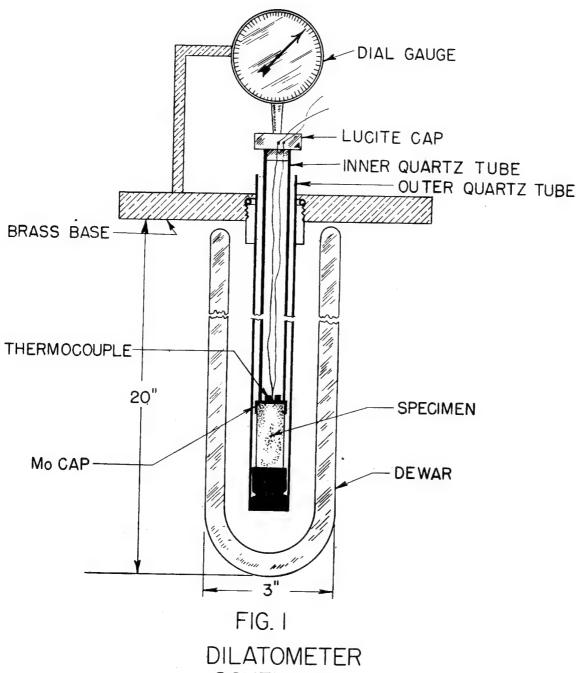
TABLE X

## Alloys

Material	T min	Method	Reference
Steels and Irons: (5) Misc. (8) 0.06 to 1.38% C Steel 1.1% C Steel 301, 304, 316, 347] 310, 330 Stainless] 304, 410, 1020 Steels	82 93 90 89 15	DIL INT DIL DIL	Henning <sup>26</sup> Dorsey III <sup>12</sup> Simon and Bergmann <sup>61</sup> Furman <sup>19</sup> O. S. U. <sup>2</sup> (App. A)
Cu - Ni alloys: (17) Complete System (13) Complete System Constantan Monel	20 77 82 15	DIL DIL INT	Krupkowski <sup>42</sup> and De Haas <sup>41</sup> Aoyama and Ito <sup>4</sup> Henning <sup>26</sup> O. S. U. <sup>2</sup> (App. A)
Others: Brass " Bronze Contracid Inconel Invar (2) " Soft Solder Fe - Ni Fe - Co - Cr (10) Pt - Ir (80-20) Pt - Ir (90-10)	82 15 82 15 15 82 82 93 83 83 82 82	DIL INT DIL INT INT INT INT DIL DIL DIL	Henning <sup>26</sup> O. S. U. <sup>2</sup> (App. A) Henning <sup>26</sup> O. S. U. <sup>2</sup> (App. A) O. S. U. <sup>2</sup> (App. A) Scheel <sup>57</sup> Valentiner and Wallot <sup>66</sup> Dorsey Il <sup>0</sup> Masumoto <sup>46</sup> Masumoto <sup>46</sup> Henning <sup>26</sup> Scheel <sup>57</sup>

TABLE XI
Inorganic Compounds

Formula	T min (°K)	Meth <b>o</b> d	Reference
Li, Na, K-F, Cl, Br, I Rb-Cl, Br, I NaCl NaF LiF LiH	89 20 85 20 20 20	DEN DEN DIL INT DIL INT DIL	Henglein I, 24 II <sup>25</sup> Henglein II <sup>25</sup> Lindemann II <sup>45</sup> Buffington and Latimer <sup>7</sup> This report p. 21 Adenstedt <sup>1</sup> This report p. 20
NH4C1 NH4C1 NH4Br (NH4)3PO4	78 200 197 150	INT DIL DIL DIL	Adenstedt <sup>1</sup> Simon and Bergmann <sup>6</sup> " " "
CaF <sub>2</sub> CaCO <sub>3</sub> (Calcite) CaCO <sub>3</sub> (Aragonite)	81 20 20	INT INT INT	Valentiner and Wallot <sup>66</sup> Adenstedt1
Mn0 MnS Fe0 Fe304 Fe304 FeS2 ZnS	105 130 160 105 88 98 20	Xray Xray Xray Xray DIL INT INT	Ellefson and Taylor <sup>15</sup> """"  """  Okamura <sup>50</sup> Valentiner and Wallot <sup>66</sup> Adenstedt <sup>1</sup>
B <sub>2</sub> O <sub>3</sub> CO <sub>2</sub> H <sub>2</sub> O	20 84 84	DIL DEN DEN	Lindemann II <sup>45</sup> Dewar <sup>8</sup>
Quartz Crystal """ """ Beryl Crystal (3Be0•Al <sub>2</sub> O <sub>3</sub> •6SiO <sub>2</sub> )	93 20 85 83 57	INT DIL INT INT INT	Dorsey II <sup>11</sup> Lindemann II <sup>45</sup> Buffington and Latimer <sup>7</sup> Nix and MacNair <sup>48</sup> Erfling II <sup>16</sup>



**SCHEMATIC** 

